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Institute of
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N-M-R
Newsletter

No. 115
APRIL, 1968

OVER

Deadline Dates: No. 116: 1 May 1968
No. 117: 5 June 1968 } PLEASE NOTE!

Reminder: For the period August 10, 1967 to August 15, 1968 inclusive, all Newsletter contributions, enquiries, etc., should be addressed as follows:

Dr. Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

- CONTINUED ON THE OUTSIDE BACK COVER

Imperial Chemical Industries Limited

PLASTICS DIVISION

Bessemer Road, Welwyn Garden City, Hertfordshire

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Professor Bernard L. Shapiro,
Department of Chemistry,
Stanford University,
Stanford,
California, 94305,
U.S.A.

Your Ref.

Our Ref. MEAC/AB/SMW/RES/2.G.

4th March, 1968

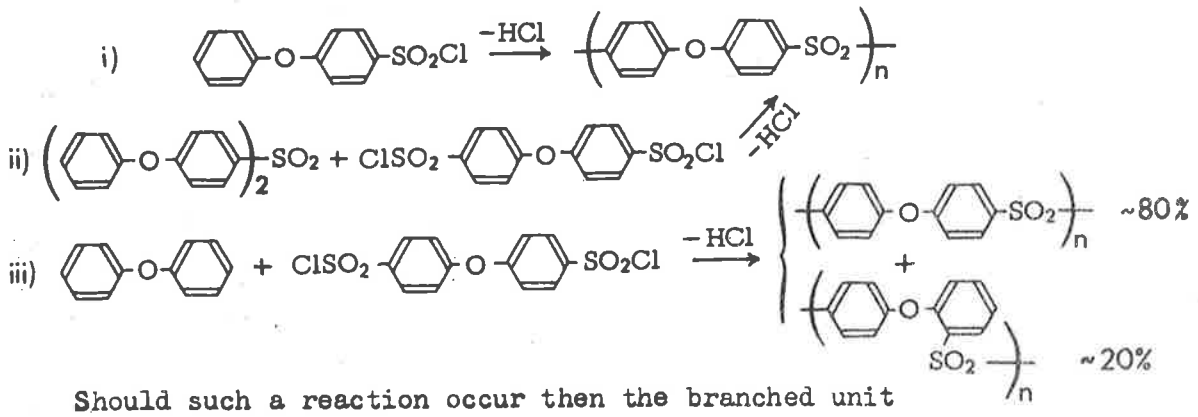
Dear Professor Shapiro,

Please accept our apologies for the delay in submitting our contribution to the newsletter.

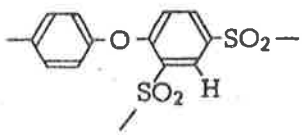
The Use of Spectrum Accumulation in the Study of Polymers

Only limited use seems to have been made of this technique in the study of polymer molecular structure. We should therefore like to report some results that we have obtained in our study of polyarylene sulphones.

It has previously been reported (Ref. 1) that the structure of polymers prepared by different Friedel-Crafts polysulphonylation reactions is dependent upon the structure of the monomers. The n.m.r. spectra of polymers prepared by the following routes have been examined in order to investigate the possible presence of branched units in such polymers due to the attack of sulphonyl chloride chain ends on either of the linear repeat units shown.



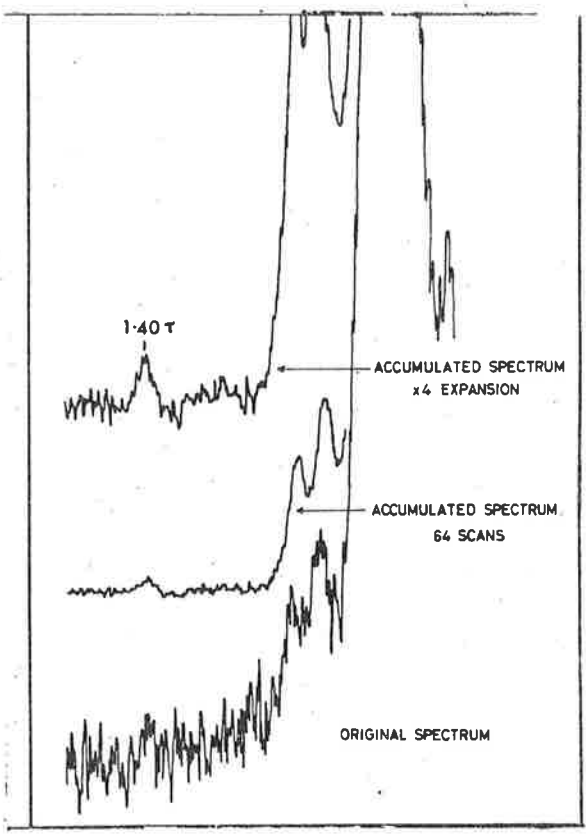
Should such a reaction occur then the branched unit



will arise.

/..

Again as previously reported, the n.m.r. spectrum should show a resonance near 1.4τ due to the proton situated between two sulphone groups as indicated. The n.m.r. spectrum of the polymers prepared from reactions (i) (iii) showed no evidence of such a resonance even under high amlification. A spectrum accumulation experiment was devised which resulted in the observation of a resonance at 1.4τ after the spectrum had been scanned a large number of times.



It was also possible to estimate the number of branches present in the polymer after calibration with known amounts of a highly branched, specially prepared polymer where the ratio of total protons to low field (1.4τ) protons was measured directly.

The results of this work compared very favourably with a chemical method of analysis and showed that the extent to which these polymers are branched depends upon both the reactants from which the polymers are prepared and the molecular weight.

Polymer from reaction	R.V.	Branches per 100 polymer repeat units
(i)	0.47	$<$ 0.4
(ii)	0.49	0.4 - 0.7
(iii)	0.11	0.7
"	0.40	1.3
"	0.55	1.7
"	0.56	1.6

/..

The accumulation experiment was performed with a J.E.O.L. RAI Spectrum accumulator in conjunction with a Varian H.A.100 n.m.r. spectrometer. All the samples and calibration standards were dissolved in dimethyl sulphoxide to give a 3.6 % wt/vol concentration. A more detailed account of this work will be published later this year in 'Polymer'.

Yours sincerely,

M.E.A. Cudby

M.E.A. CUDBY
Research Department

A. Bunn

A. BUNN
Research Department

Ref. M.E.A. Cudby, R.G. Feasey, B.E. Jennings, M.E.B. Jones and J.B. Rose
Polymer, Vol. 6, p 589, 1965

AMERICAN OIL COMPANY

RESEARCH AND DEVELOPMENT DEPARTMENT 2500 NEW YORK AVENUE WHITING, INDIANA 46394

March 4, 1968

Professor Bernard L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

Dear Dr. Shapiro:

Please add us to your list of subscribers of the most informative and useful "N.M.R. Newsletter". We believe the following should qualify us for a subscription.

V-4341 VARIABLE TEMPERATURE SYSTEM MALFUNCTION

This is the standard Varian temperature system used on the HA-60-IL, DA-60-IL and the HA-100 and consists of the V-4343 Temperature Controller, stainless steel heat exchanger coil, polystyrene Dewar, and with the heater-sensor in the V-4333 probe. The problem encountered was the system not regulating the desired temperature which was in the range from +5°C to +20°C (i.e., just a little below room temperature). The symptoms of malfunction were: 1) slow oscillation of the needle on the REGULATION RANGE meter, at best, and at worst the needle at one end of scale moving to the other end of scale back and forth; 2) a random shifting of the methanol peak from one scan after another at 50 Hz SWEEP WIDTH on the paper. The remedy in our case was replacement of the Zener diode in the V-4343 temperature controller unit. We tried, without rectifying the situation, replacing all Tygon tubing, using dry ice instead of liquid air in the heat exchanger, and rotation of the heater-sensor in the probe.

One recurrent, often repeated theory as to the cause of the above mentioned symptoms, deserves comment. This theory states that the gaseous dry nitrogen flowing at 10-15 SCFH causes the heater-sensor to flop around or vibrate inside the probe. We constructed an unsilvered vacuum jacketed extension, 30 cm. long and just large enough to house the heater-sensor, so that we could observe what was occurring to cause oscillation of the REGULATION RANGE needle. Without liquid air in the heat exchanger or the V-4343 unit ON, dry nitrogen flowing as high as 15 SCFH caused no visible vibration of the heater-sensor element. With liquid air in the heat exchanger and the V-4343 ON and set at ca. +10°C, the REGULATION RANGE needle did slowly oscillate. Quite clearly, the meter needle movement corresponded with actual movement of the heater-sensor. Heater-sensor movement was caused by a cold blast of air caus-

ing the exposed resistence wire leads (going to the heater part) to contract, thus moving the heater-sensor to the center of air flow. On the heating cycle, the resistence wire leads expanded causing the heater-sensor to bend toward one side of the air flow. Rotation of the heater-sensor while the temperature control system was in operation did cause the thing to quiet down, that is, the needle remained in one spot of REGULATION RANGE and the methanol peak was superimposed after successive scans. In conclusion then, we feel the "air flow vibration theory" is incorrect as air flow alone does not cause the heater-sensor to rattle around.

L&N GALVANOMETER OF THE VK-3506 FLUX STABILIZER

The characteristic behavior of galvo trouble is as follows: 1) slight oscillation when centering hairline in BALANCE, 2) galvo kicks off when going to OPERATE necessitating several attempts to lock, 3) going from OPERATE back to BALANCE the galvo oscillates back and forth for longer than one minute (usually about five or more), and 4) HA-60-IL and HA-100 lose field-frequency lock very easily resulting in the STABILIZER kicking out. Characteristics 2) and 3) are the most obvious and also the most annoying. This trouble is due to lack of damping in the Leeds & Northrup Galvanometer Movement. Over a period of time, a few years, the damping mechanism of the galvo wears out. These movements can be purchased new for $91 (or the old one exchanged at a lower price) from your local L&N dealer. Specify part No. PI-195D for Model 2430D Leeds & Northrup Galvanometer, when ordering. Be sure to emphasize "D" as this designates the necessary sensitivity for the VK-3506 Field Flux Stabilizer.

Sincerely,

Bruce E. Wenzel

BRUCE E. WENZEL

Em Banas

E. M. BANAS

BEW:EMB/crh

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Your Ref.

Our Ref. JKB/AR

Dr. B.L. Shapiro,
Chemistry Department,
Stanford University,
Stanford,
California 94305,
U.S.A.

Dear Barry,

Calculation of Solvent Magnetic Effects

A useful feature of 220 Mc/s proton nmr is that solvent effects on chemical shifts are proportionately larger than at lower frequencies. This means that the number of instances where differential solvent shifts can be used to separate overlapping resonances is increased. Several organic chemistry laboratories are currently interested in using solvent shifts for structure diagnosis [e.g. J. Ronayne and D.H. Williams, J. Chem. Soc. (B), 540 (1967); J. Chem. Soc. (C), 2642 (1967); F. Hruska, D.W. McBride and T. Schaefer, Canad. J. Chem., 45, 1081 (1967); T. Winkler and W. von Philipsborn, Helv. Chim. Acta, 51, 183 (1968)]. We share this interest, and to this end we are doing both theoretical and experimental work. I mention here a calculation of solvent anisotropy shifts for non-polar spherical solute molecules in solvents with axially symmetric molecules; whilst it applies strictly only for this special case, it gives some insight into solvent anisotropy effects generally. For example, it provides a qualitative explanation of the correlation with molecular radius reported recently by Winkler and von Philipsborn for methyl-substituted cycloalkanes.

The most important features resulting from the calculation (for a spherical solute molecule with no strongly polar groups) are:

(i) as a result of random tumbling, the anisotropy contribution σ_a to the nuclear screening is independent of the position of the nucleus within the solute molecule, being equal to that calculated for a nucleus at the centre of the molecule;

(ii) $|\sigma_a|$ decreases with increasing solute molecule radius.

The principal assumption made is that the solute and solvent molecules move randomly subject to the limitations imposed by their surfaces of

Cont/.....

FROM: J.K. Becconsall

SHEET NO.: 2

TO: Dr. B.L. Shapiro

DATE: 11.2.68.

exclusion. For a given molecule the surface of exclusion must approximate closely to the envelope of van der Waals radii. The forms used are:

for a disc-shaped molecule (e.g. benzene), a cylindrical middle section with edges of semicircular profile;

for a rod-shaped molecule (e.g. carbon disulphide), a cylinder with hemispherical caps at its ends.

The expressions obtained for σ_a are cumbersome, and in the case of disc-shaped solvent molecules the evaluation calls for a numerical integration. They satisfactorily predict the order of magnitude of the anisotropy shift and its dependence on solute molecule radius. The differential solvent shifts measured for methane, tetramethylsilane and cyclohexane at low solution concentrations in benzene and in carbon tetrachloride are, however, about half of the predicted shifts.

For spherical solute molecules at least, it should be possible to improve the model by including the effects of attractive van der Waals forces, which are expected to reduce $|\sigma_a|$ by pulling the extremities of the solvent molecule towards the surface of the solute molecule. I also anticipate an extension to non-spherical solute molecules, although this is likely to call for some different mathematical techniques. The work done up to now has been submitted for publication in Molecular Physics, and some preprints should be available in due course for anyone interested.

In any work involving external referencing using the Varian HR-220 spectrometer, it must be remembered that the magnetic field is parallel to the axis of the sample tube; the well-known expression $\sigma_b = (2/3)\pi\chi$ for the bulk susceptibility effect in a conventional spectrometer is in this case replaced by $\sigma_b = -(4/3)\pi\chi$. This difference suggests an obvious and very convenient method of measuring diamagnetic susceptibilities for any laboratory having both a superconducting solenoid spectrometer and a conventional spectrometer, using an ordinary coaxial sample cell. Unfortunately in our case we cannot do it without changing sample tubes, as our Perkin-Elmer R.10 requires 4.6 mm. o.d. tubes.

Best wishes,

Jack Becconsall

J.K. Becconsall

115-8

Dr. Ludwig Pohl
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c/o E. MERCK · DARMSTADT
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Herrn

Professor Dr. B.L. SHAPIRO
Department of Chemistry
Illinois Institute of Technologie

C h i c a g o, Illinois 60616

U.S.A.

Ihre Zeichen	Ihr Schreiben vom	Hausruf	Unsere Zeichen	DARMSTADT
			Dr.Po/Dr.Meh/Sch	1. März 1968

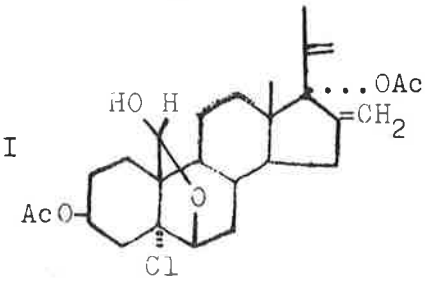
Betr.: Analyse C-19-anomerer Steroidgemische

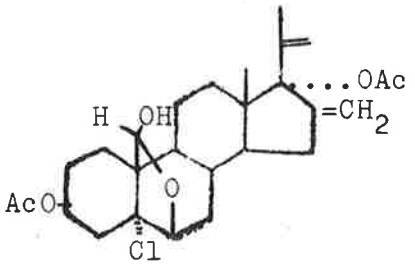
Sehr geehrter Herr Professor Shapiro!

Im Rahmen einer Arbeit über Synthesewege zum 16-Methylen-17α-hydroxy-19-norprogesteron und seinen Derivaten, die in Kürze erscheint, wurden bei der C-19-Funktionalisierung eine Reihe von 19S- und 19R-Anomeren hergestellt, u.a. auch folgende zwei Verbindungen:

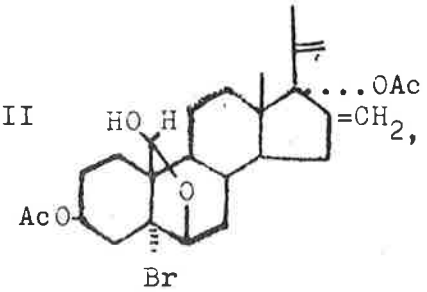
19S- 19R-

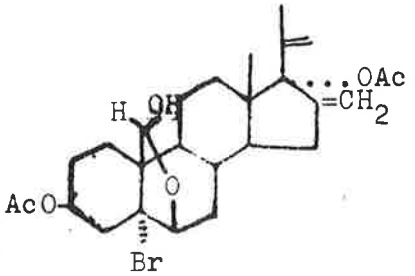
I





II





Es zeigt sich dabei, dass derartige Anomere sowohl in $CDCl_3$ als auch in d_6-DMSO bezüglich der chemischen Verschiebung von C-18 und C-19 Unterschiede aufweisen, die in $CDCl_3$ grösser als in d_6-DMSO sind. In der Reihe der von uns untersuchten Anomeren lagen die $\Delta\delta$-Werte für C-18 zwischen o,o2 und o,o5 ppm, für 19-OH bei o,2o ppm und für 19-H zwischen o,4 und 1,oo ppm. Ein Einfluss auf andere Signale ist nicht zu beobachten.

Da im 19S-Anomeren die entschirmende 19-OH-Gruppe weiter von C-18 entfernt ist als im entsprechenden 19R-Anomeren, muss das bei höherem Feld liegende Signal dem S-Anomeren zugeordnet werden. Eine analoge Betrachtung über die Wechselwirkung zwischen C-19 und C-3 führt zur entsprechenden Zuordnung der 19-H-Signale und zur entgegengesetzten der 19-OH-Signale.

	C-18		19-H		19-OH	
Subst.	R	S	R	S	R	S
I	o,62	o,6o	5,55d	5,15d	6,72d	6,87d
			j = 4,5 Hz		j = 4,5 Hz	
II	o,62	o,6o	5,58d	5,o5d	6,72d	6,87d
			j = 4,5 Hz		j = 4.5 Hz	

Lösungsmittel: d_6-DMSO

Bei der präparativen Aufarbeitung von C-19-Anomerengemischen können daher die einzelnen Anomeren auch quantitativ leicht NMR-spektroskopisch identifiziert werden.

Vgl.: M.E. Wolff, S.Y. Cheng, J.org.Chem. 32, 1o29 (1967),
S. Hara, K. Oka, Y. Ike, Chem.and Ind. (1967) S.832.

Mit freundlichen Grüssen

L. Kohl W. Mehrhof

IMPERIAL CHEMICAL INDUSTRIES LIMITED

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Professor B.L. Shapiro,
Department of Chemistry,
Stanford University,
Stanford,
California.

Your ref:

Our ref: JSG/SB

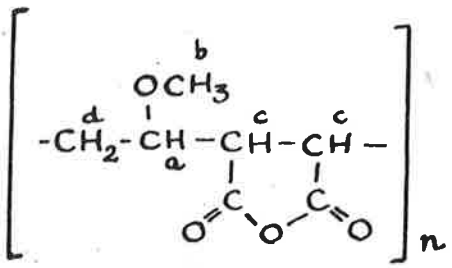
Date: March, 1968.

Dear Professor Shapiro,

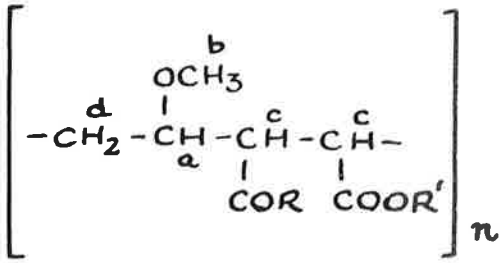
Copolymers of Methyl Vinyl Ether with Maleic anhydride and Substituted Maleic Acids

Thank you for the reminder and please excuse the delay of our contribution.

We have recently prepared a series of copolymers of methyl vinyl ether with maleic anhydride and with free and substituted maleic acids as shown in I and II.



(I)



(II)

Our main problem has been to determine the degree of either hydrolysis or esterification of I to form copolymers of type II under various conditions. Since most of these compounds are soluble only in aqueous media, infra-red techniques have proved difficult and accordingly we have examined them by NMR using a Perkin-Elmer R10 60 Mcs spectrometer.

FROM: J.S. Glasby

CONTINUATION SHEET NO.: 1

TO: Professor B.L. Shapiro

DATE: March, 1968.

In Table 1 are given the assignments for the various protons, those due to -COOH, $-NH_2$ and $-NH_4^+$ not being observable due to exchange with D^+ since all of the compounds were examined in D_2O or acetone $-D_6$.

Table 1

Compound	a	b	c	d
I	5.8	6.4	6.4	7.7
II R=OH, R'=H	6.4	6.7	7.1	8.1
II $R=NH_2$, $R'=NH_4^+$	5.6	5.9	6.5	7.4
II R=OH, $R'=Na^+$	5.4	5.7	6.3	7.2
II R=OH, R'=Et	6.3	6.6	6.9	7.9
II R=OH, $R'=Pr^i$	6.2	6.6	6.9	7.9
II R=OH, R'=Bu	6.4	6.7	7.1	7.9

All of the above are τ-values.

The Table shows that both hydrolysis to the free acid and esterification produce a general shift to higher field and in particular, the protons α to the carboxyls can be well separated from the methoxyl protons. Salt formation, on the other hand, produces a similar shift downfield.

Curiously, we have found no difference between the two α-protons (c) even in those compounds having different substituents on these carbon atoms.

On the basis of these results we have been able to estimate the approximate degree of hydrolysis or esterification in the mixed copolymer.

Yours sincerely,

John S. Glasby

(J.S. Glasby)

115-12

RUKSUNIVERSITEIT-GENT

LABORATORIUM
voor
ORGANISCHE CHEMIE
M.Verzele
Dir.: Prof. Dr. ~~F. GOVAERT~~

GENT, March 12th 1968.

J. Plateaustraat, 22
België - Europa
Tel. 23.38.21

Assoc. Prof. B. L. SHAPIRO,
Department of Chemistry,
Stanford University,
STANFORD. CALIFORNIA 94305.

Unpredictable Shifts.

Dear Prof. Shapiro,

We have shown earlier that in 4,6-disubstituted m-dioxanes an empirical relationship exists between $\delta_{2e \to 2a}$ and the degree of branching of the alkyl substituents. This is not only so for cis derivatives (1), but also for the trans series (2). Computation of shift values for whatever compound however remains difficult, because the midpoint of the AB system of the H-2 protons also depends slightly on the nature of substitution. In 4,6-dialkyl derivatives however, this complex problem may be solved (2).

We have now measured a lot of derivatives of m-dioxane, having also polar groups, i.e. CF_3 and Ø... Some of the results are shown in the table. The influence either on H-2e or H-2a for a 4-eq. Ø group f.i. falls fairly well into a monotonous trend as the other substituent is changed (Me → t.Bu) : no 1,2 (cf. 3 for the introduction of 4-eq. Me). However, this is not true for a 4-eq.CF_3 group : no 4 and 5. Such deviations from pure "additivity" of shifts seem even to be more pronounced for axial groups. Although it is perhaps obvious for reasons of ring deformation that the values found in 6 and 7 are not comparable, this is not so for 7 against 9. It is remarkable however that for non polar combinations (compare 7 and 8), the agreement is not too bad. (All these compounds are enancomeric (biased) and occur in chair forms, as follows from the observed coupling values (3)). Also the comparison for entries 12 and 13 (non-enancomeric) is bad, although the similarity of the $\delta_{2e \to 2a}$ values indicate the equilibrium state being for both roughly the same. Perhaps the most drammatic non-conformity may be found in entries 14, 15 and 16. Again the difference between 14 and 15 is perhaps trivial (gem Ø substitution; -hence rotamer-changes ?), but this is not for 15 and 16.

From all these examples and others it follows that it is extremely difficult to predict exact shift values by comparing with standard values obtained from model compounds, especially when one of the substituents is polar and axial in nature. Presumably a special solvation effect is at the basis of the capricious changes ? None of other criteria(dipole moment

.../...

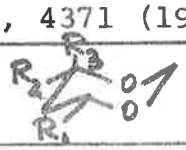
changes (4), concentration dependence or solvent bulk susceptibility...) may be at the basis of the variation. Presumably the introduction of an extra group may alter the specificity of the solvation shell which existed before the introduction of this group, and this should especially be true for polar derivatives (polar substituents and "polar"=axial positions). Some of these results will be published in more detail in the near future.

Truly yours,

Prof. M. Anteunis.

(1) D.Tavernier, M.Anteunis; Tetrah.Letters 47, 5851 (1966).
(2) D.Tavernier, M.Anteunis; unpublished results.
(3) D.Tavernier, M.Anteunis; Bull.Soc.Chim.Belges 76, 157 (1967).
(4) J.Delmau, J.-C.Duplan, H.Davidson; Tetrahedron 23, 4371 (1967).

Relative Shifts for H-2e and H-2a in 1,3-dioxanes[a] (Shift differences of compounds I versus II).



n°	I trans	I cis		II trans	II cis		ΔH-2e	ΔH-2a
	R_1	R_2	R_3	R_1	R_2	R_3		
1)	Ø	Ø	H	Ø	H	H	-22.39	-20.03
2)	Me	Ø	H	Me	H	H	-23.93	-20.90
	Et	Ø	H	Et	H	H	-23.70	-21.42
	i.Pr	Ø	H	i.Pr	H	H	-23.70	-21.31
	t.Bu	Ø	H	t.Bu	H	H	-24.72	-21.78
3)	Me	Me	H	Me	H	H	- 1.50	- 0.02
	t.Bu	Me	H	t.Bu	H	H	- 1.51	+ 0.23
	Et	Me	H	Et	H	H	- 1.59	- 0.03
	Ø	Me	H	Ø	H	H	- 1.55	- 0.55
4)	t.Bu	CF_3	H	t.Bu	H	H	-19.69	- 5.96
5)	Ø	CF_3	H	Ø	H	H	-10.24	- 2.12
6)	t.Bu	H	Me	t.Bu	H	H	+24.24	-30.36
7)	Me	Me	Me	Me	Me	H	+20.14	-22.17
8)	t.Bu	Me	Me	t.Bu	Me	H	+21.47	-22.03
9)	Ø	Me	Me	Ø	Me	H	+11.22	-13.35
10)	t.Bu	H	CF_3	t.Bu	H	H	+ 5.61	-40.62
11)	Ø	H	CF_3	Ø	H	H	+16.89	-30.94[b]
12)	H	Ø	Me	H	H	H	- 7.06	+ 7.88[c]
13)	Ø	H	Me	H	H	H	-19.82	- 6.64[d]
14)	t.Bu	H	Ø	t.Bu	H	H	+10.73	-15.35
15)	Ø	Ø	Ø	Ø	Ø	H	+14.63	-19.32
16)	t.Bu	Ø	Ø	t.Bu	Ø	H	+15.26	+ 3.48
17)	Ø	H	Ø	Ø	H	H	+17.07	-15.56

a) 0.85 mmol of product + 0.11 mmol 1,3-dioxane as internal reference in 1 ml CS_2; in cps under standard conditions ; at 100 Mc. Shifts are positive when at increased field position.

b) trans-4-Ø-6-CF_3-m.dioxane seems to be conformationally pure with CF_3 axial (from J-values).

c) $\Delta\delta_{2e \rightarrow 2a}$ = 14.94 cps.

d) $\Delta\delta_{2e \rightarrow 2a}$ = 13.18 cps.

THE OHIO STATE UNIVERSITY
DEPARTMENT OF CHEMISTRY
88 WEST 18TH AVENUE
COLUMBUS, OHIO 43210

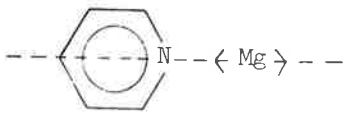
March 13, 1968

Dr. Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

T_1 of N^{14} and exchange rates

Dear Barry:

As I pointed out some time ago in the newsletter, the nmr linewidths for the α-protons in pyridine are quite dependent on the nature of the solvent, on hydrogen bonding and metal complexation, respectively at nitrogen. Pure pyridine in ether, or the pure pyridine·dibutylmagnesium complex gives the usual broad lines for the α-protons. However mixtures of these two species in ether give time averaged spectra, the α-proton lines of which are much too narrow to be accounted for by just fast nitrogen metal exchange. Since the broadening observed, first, must come from some degree of nitrogen-proton coupling, its absence indicates the operation of a much shorter T_1 for nitrogen than is normal for pyridine (Moniz and Gutowsky) or its complex. What we would like to suggest is that fast nitrogen magnesium exchange is responsible for lowering the T_1 of nitrogen. Fast exchange should produce rapid fluctuations of the electric field gradient, around, $\frac{\delta^2 F}{\delta z^2}$, nitrogen. This fluctuation will not average out the field gradient because most likely the attack and dissociation of the magnesium takes place along the principal molecular axis, i.e. the motion of the



magnesium with respect to the nitrogen is anisotropic.

We are presently observing the above effect in benzisoquinoline and trying to match the α-proton lineshapes with calculated ones for various rates of chemical exchange and T_1 relaxation. By assuming the N^{14}, α-proton coupling constant (corresponding N^{15}-proton coupling constants are known in similar heterocycles) and the shifts for the various species we should be able to estimate the exchange rates and T_1s.

With best regards.

Sincerely yours,

Gideon

Gideon Fraenkel
Associate Professor
of Chemistry

UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF MINES
4800 FORBES AVENUE
PITTSBURGH, PENNSYLVANIA 15213

March 19, 1968

Pittsburgh
Coal Research Center

Dr. Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

Dear Barry:

MAGNETIC RELAXATION IN BITUMINOUS COAL AND AMORPHOUS CARBON

Room temperature nuclear magnetic relaxation times for the protons in vitrain-rich Pittsburgh Seam coal (hvab; 82.6% m.a.f. carbon) and the ^{13}C nuclei in an amorphous carbon prepared by the 1000°C reduction of $^{13}CO_2$ have been measured through the generous co-operation of NMR Specialties, Inc.

T_1 and T_2 were found to be 50 μsec. and 9 μsec. respectively for the protons in the coal. Since all coals give strong ESR signals, it is likely that the rapid spin-lattice relaxation is due to the presence of paramagnetic species, presumably free radicals, in the coal.

T_1 and T_2 measurements for the ^{13}C nuclei in the amorphous carbon which contained 60% ^{13}C gave values of 36 msec. and 80 μsec. respectively. This material also gives an ESR signal; no evidence for electron-nuclear hyperfine splittings was found. Broadline NMR studies of the carbon are in progress.

Sincerely yours,

Herb

H. L. Retcofsky

R. A. Friedel

115-16
INDEPENDENCE MALL WEST PHILADELPHIA, PA. 19105 TELEPHONE (215) 592-3000

REPLY TO:
ROHM AND HAAS COMPANY
REDSTONE RESEARCH LABORATORIES
HUNTSVILLE, ALA. 35807

ROHM AND HAAS COMPANY

March 15, 1968

Dr. Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

Dear Barry,

The existence of rotameric forms of tetrafluorohydrazine (N_2F_4) was shown by the F^{19} NMR at -155°C (J. Chem. Phys., 43, 4526); about equal amounts of 'trans' and a d, ℓ 'gauche' pair were found. An electron diffraction study, however, finds only 'gauche' (R. K. Bohn and S. H. Bauer, Inorg. Chem., 6, 304), while independent Raman-IR studies (J. R. Durig, F. A. Miller, private communications to C. B. Colburn) find both 'trans' and 'gauche'.

We were encouraged by this interest to improve upon our earlier F^{19} work, and did so mainly by using solvent mixtures rich in NF_2NO and NO. An experimental half-spectrum of the 'gauche' rotamer is shown in Fig. 1 - the 'trans' isomer has only a single line.

The 'gauche' spectrum is interpretable as an AA'BB' system with the parameters given in Table I, derived from iteration with the Swalen-Reilly NMRIT program. The calculated spectrum is shown in Fig. I. No deviation greater than 1.0 cps occurs - satisfactory, since the standard deviation of measured line positions is 1.4 cps (a combination of field drift and fat lines). While the magnitudes of the coupling constants are reasonable, the change in sign of the vicinal J's with dihedral angle has no precedent of which I am aware.

Sincerely,

Fred

F. A. Johnson
(205) 876-9037

FAJ:mm

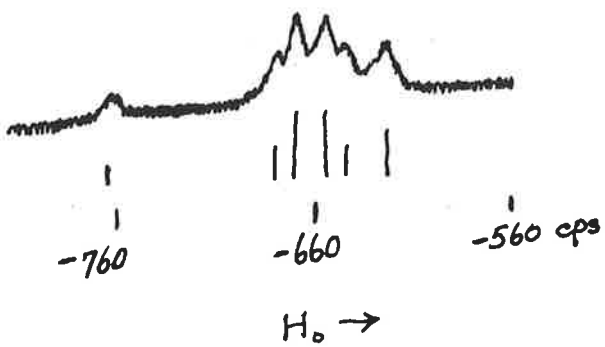
Enclosures

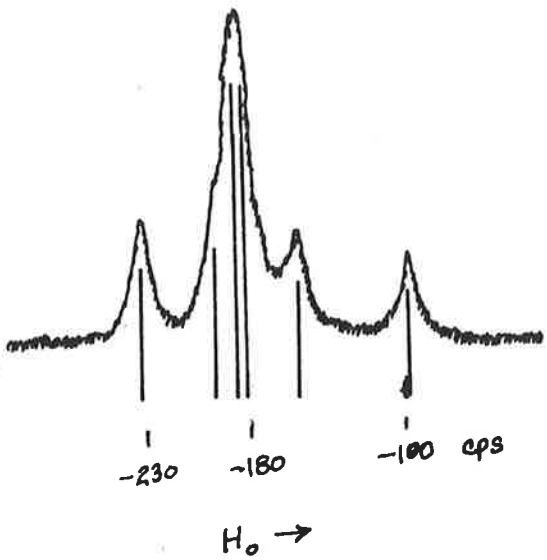
TABLE I

$\delta = 688.6 \pm 0.1$	$J_{13} = \mp 13.4 \pm 0.2$
$J_{14} = \pm 489.6 \pm 0.2$	$J_{34} = \pm 109.0 \pm 0.2$
$J_{12} = \mp 58.8 \pm 0.2$	$H_0(1,2) < H_0(3,4)$

FIG. I

$F_1 + F_2$ at -344.3 cps





115-18

UNIVERSITY OF ILLINOIS
Department of
CHEMISTRY AND CHEMICAL ENGINEERING
URBANA
61801

The William Albert Noyes Laboratory

UNIVERSITY OF ILLINOIS CENTENNIAL YEAR 1967-68

March 25, 1968

Title: The Dissociation of Lithium and Sodium Tetramethylaluminate in Ether, Tetrahydrofuran, and 1,2-Dimethoxyethane.

Dr. Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

Dear Barry:

We have just completed an investigation of the dissociation of lithium and sodium tetramethylaluminate (LiTMA and NaTMA) in the solvents ether, tetrahydrofuran (THF), and 1,2-dimethoxyethane (DME).

Our results for the ether and DME solutions are in agreement with those of other workers [1,2]. In ether the two salts exist predominantly in the intimate ion pair form and in DME, in the solvent separated ion pair form.

However in THF it was found that significant quantities of both types of ion pairs exist. The proton lineshape of the salts varies considerably with temperature and concentration. We have analyzed the lineshapes assuming that the exchange rate between the two species is fast enough so that the spectral parameters obtained from the experimental spectra are the weighted averages of the parameters for each of the two sites. Thus, the exchange process governing the proton lineshape is the spin-lattice relaxation of the ^{27}Aℓ nucleus, which tends to average out the Aℓ-H J-coupling.

It was found that the plot of R_1 ($R_1=1/T_1$) of the Aℓ nucleus versus temperature has a minimum in the case of LiTMA. For NaTMA no minimum was observed but R_1 was noted to increase with increasing temperature. This is a clear indication that R_1 cannot be determined solely by the quadrupole relaxation of a single molecular species, for if this were the case, R_1 would decrease monotonically with increasing temperature. In other words, the temperature dependence of R_1 reflects the changing equilibrium

proportions of the intimate and solvent separated ion pair forms as well as the temperature dependence of the correlation times for their reorientation.

From the variation of R_1 with temperature we were able to estimate some of the thermodynamic functions for the THF systems at 40°C. Writing the equilibrium as:

$$MA\ell\ (CH_3)_4 \rightleftarrows M^+ || A\ell(CH_3)_4^-$$

then for the LiTMA-THF system, K=110, ΔH^o =-6·5 kcal/mole, and ΔS^o=- 11 e.u. Only K could be determined for the NaTMA-THF system and this was 11.

Combining the proton lineshape data with some $^{27}A\ell$ resonance data we were able to calculate the $A\ell$-CH_3 coupling constants for various species. For the intimate ion pair of LiTMA J=7·1 Hz. and for NaTMA J=9·1 Hz. J was found to be the same (within experimental error) for the solvent separated ion pair resulting from each salt and was equal to 6·3 Hz.

Sincerely yours,

E. S. Gore H. S. Gutowsky

[1] K. C. Williams and T. L. Brown, J. Amer. Chem. Soc., 88,4134 (1966).
[2] J. P. Oliver and C. A. Nilkie, J. Amer. Chem. Soc., 89,163 (1967).

CHEMISCHES LABORATORIUM
DER UNIVERSITÄT MÜNCHEN
INSTITUT FÜR ORGANISCHE CHEMIE

8000 MÜNCHEN 2,
Karlstr. 23 - Tel. 557976

March 21, 1968

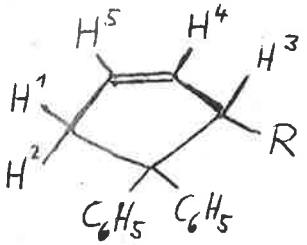
Professor B.L.Shapiro
Department of Chemistry
Stanford University
Stanford, Calif. 94305
USA

Dear Professor Shapiro:

"Cyclopentene Spectra involving Long-Range Coupling"

Since Professor Zimmermann and his group have left for Freiburg, we faced the unpleasant experience to live without your "Newsletters". We feel that somebody in the Munich institute should be on your mailing list, so I hope that this might go as my first contribution.

I had the opportunity to look at the spectra of some cyclopentenes (general structure 1 shown below) which have been synthesized by Dr. Dahl in Professor Huisgen's group. Each line of the AB system formed by the protons 1 and 2 in 1a and 1b shows up as a nearly binomial quartet; proton 3 appears as a quintet, regular in 1b, distorted in 1a. Compound 1c has a more complicated but still similar spectrum, whereas the spectrum of 1d consists of only two sharp lines, intensity ratio 1:2.



1

These ABMXY-type spectra could be simulated with a modified version of the improved Marquardt-Ferguson program described by J.D.Roberts and F.J.Weigert in these letters.

The chemical shifts ν_i (in Hz at 60 MHz downfield from internal TMS) and couplings J_{ij} of the protons 1 through 5 were obtained with good convergence and are shown in the table. Although the spectra involve some virtual coupling through the olefinic protons, all solutions other than that in the table could be ruled out due to slight asymmetries in the splitting pattern of 1a, except for the interchange of protons 4 and 5 and for the signs of J_{12} and J_{45}.

Because of the deceptive simplicity of their spectra, the parameters for 1c and 1d could not be obtained by the iterative procedure, but are shown to lie along the general lines found for 1a and 1b.

Decoupling experiments would certainly be desirable to confirm these assignments, but the results already obtained should be of interest for those looking for long-range coupling in the system  for which evidence is slowly accumulating. No long-range coupling could be detected in the epoxides of coupounds 1a and 1c, the only ones investigated.

Sincerely,

Rudolf Knorr

(Rudolf Knorr)

R.Knorr

Compound	1a	1b	1c	1d
R =	CO_2CH_3	C≡N	C_6H_5	H (6)
ν_1	168.9	176.0	159	180
ν_2	211.2	201.9	205	180
ν_3	270.1	262.0	283	180
ν_4	353.3	341.5	349	343
ν_5	363.7	360.8	348	343
ν_6	-	-	-	180
J_{12}	(±)16.05	-16.76	-17	-16
J_{13}	+ 1.80	+ 2.16	+ 1	+ 2
J_{14}	- 2.17	- 2.34	- 2	- 2
J_{15}	+ 2.19	+ 2.41	+ 2	+ 2
J_{16}	-	-	-	+ 2
J_{23}	+ 1.77	+ 2.02	+ 2	+ 2
J_{24}	- 2.12	- 2.03	- 2	- 2
J_{25}	+ 2.15	+ 2.57	+ 2	+ 2
J_{26}	-	-	-	+ 2
J_{34}	+ 2.10	+ 2.17	+ 2	+ 2
J_{35}	- 2.13	- 2.31	- 2	- 2
J_{36}	-	-	-	-16
J_{45}	(±) 5.82	+ 5.83	+ 6	+ 6
J_{46}	-	-	-	+ 2
J_{56}	-	-	-	- 2

115-22

CARNEGIE-MELLON UNIVERSITY

DEPARTMENT OF CHEMISTRY - 4400 FIFTH AVENUE - PITTSBURGH, PA. 15213

OFFICE OF THE CHAIRMAN

7 March 1968

Dr. B. L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

Dear Barry:

At the 9th Experimental NMR Conference we took the opportunity to poll a number of readers of IITNMR newsletter on the degree to which they used the MELLON bibliography. The answers suggested that the number of references has become so large, that without some system of preclassification, it is too time-consuming and too unrewarding to wade through the whole list, and that in fact, very little use is being made of the bibliography now. We have considered plans for critical selection, indexing or abstracting, but any really useful plan appears to be beyond our limited resources to carry out. In view of the fact that the clerical cost in typing and listing all the references is now a sizeable fraction of our research budget, we feel that the time has come to relinquish our monopoly on the item and give some one else a chance to grapple with it, if they are so inclined.

We feel rather sad at the idea of giving up a tradition of such long standing, and we will listen to and seriously consider any vigorous protests at the discontinuance of the bibliography. However, unless something drastic happens, the accompanying bibliography will be the last one.

Le roi est mort - Vive le roi (i.e. Preston Abstracts, computer-aided literature retrieval, KWIC, etc. etc.).

Sincerely,

Alhal
A. Bothner-By
Tari
S. Castellano
M. P. Williamson
D. G. Davis
R. Sprecher Rich
J. Magnuson Jim

and many others who have joined our post-prandial periodical perusal.

Title: Sic transit bibliography.

The University of Western Ontario, London, Canada

College of Science
Department of Chemistry

March 27, 1968

Dr. B. L. Shapiro
Department of Chemistry
Stanford University
Stanford, Calif. 94305
U. S. A.

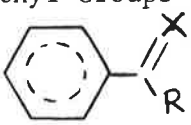
Dear Barry:

RE: ^{17}O Carbonyl Shieldings in Acetophenones

To bring my subscription up-to-date, following your blue reminder, I'd like to report some recent results obtained by Denny Sardella from the ^{17}O spectra of a few substituted acetophenones.

Some time ago, our survey of carbonyl carbon shieldings revealed that these shieldings are insensitive to m- and p-substituents in the acetophenone series [Can. J. Chem. 43, 479 (1965)] and it seemed reasonable to suppose that this is due to the fact that the carbon atom is not at the terminus of the conjugated system. Our first test of this idea was an examination of the vinyl carbon shieldings of substituted styrenes which showed that the β-carbon shielding was appreciably substituent dependent although that of the α-carbon is not [Can. J. Chem. 43, 510 (1965)]. It follows that the carbonyl oxygen in the acetophenone series should be similarly affected. Denny prepared some ^{17}O-enriched examples and their shieldings indeed exhibit the expected trend. The results are shown in the Figure vs. the β-carbon shieldings for the corresponding styrenes. In addition, the effect of ortho-methyl groups was briefly examined and the results may be summarized as follows.

Effect of ortho-Methyl Groups on the X Shieldings*

of 

Subst.	X = ^{17}O(R=Me)	$^{13}CH_2$ (R=H)
nil	-554	80.5
2-Me	-586	79.1
2,6-Me_2	-608	74.3
2,4,6-Me_3	(-595)†	74.7

* in p.p.m. $\delta_O^{H_2O}$ ± 5; $\delta_C^{CS_2}$ ± 0.3

† much less precise, ca. ± 15 p.p.m.

continued...../2

From the Table it is apparent that steric inhibition of conjugation has a pronounced deshielding effect of up to 50 ppm. on the carbonyl oxygen. This change is in qualitative agreement with the correlation of Figgis, Kidd and Nyholm, Proc. Roy. Soc. A269, 469 (1962), namely, δ_o vs. $\Delta E(n \rightarrow \pi^*)$.

The ^{17}O results were obtained at 8.13 MHz using the wide-line capability of our DP-60 instrument and the shifts were measured in acetone solutions relative to external H_2O using the concentric cell arrangement with which we have usually done our ^{13}C spectra. The ^{17}O enrichment is readily estimated from the IR spectra comparing the intensities of the $C=^{18}O$ band with the $C=^{16}O$ band, and knowing, by mass spectrometry, the $^{18}O/^{17}O$ ratio of the enriched water used for the exchange. A check by mass spectrometry established the validity of the IR method.

Best regards.

Sincerely,

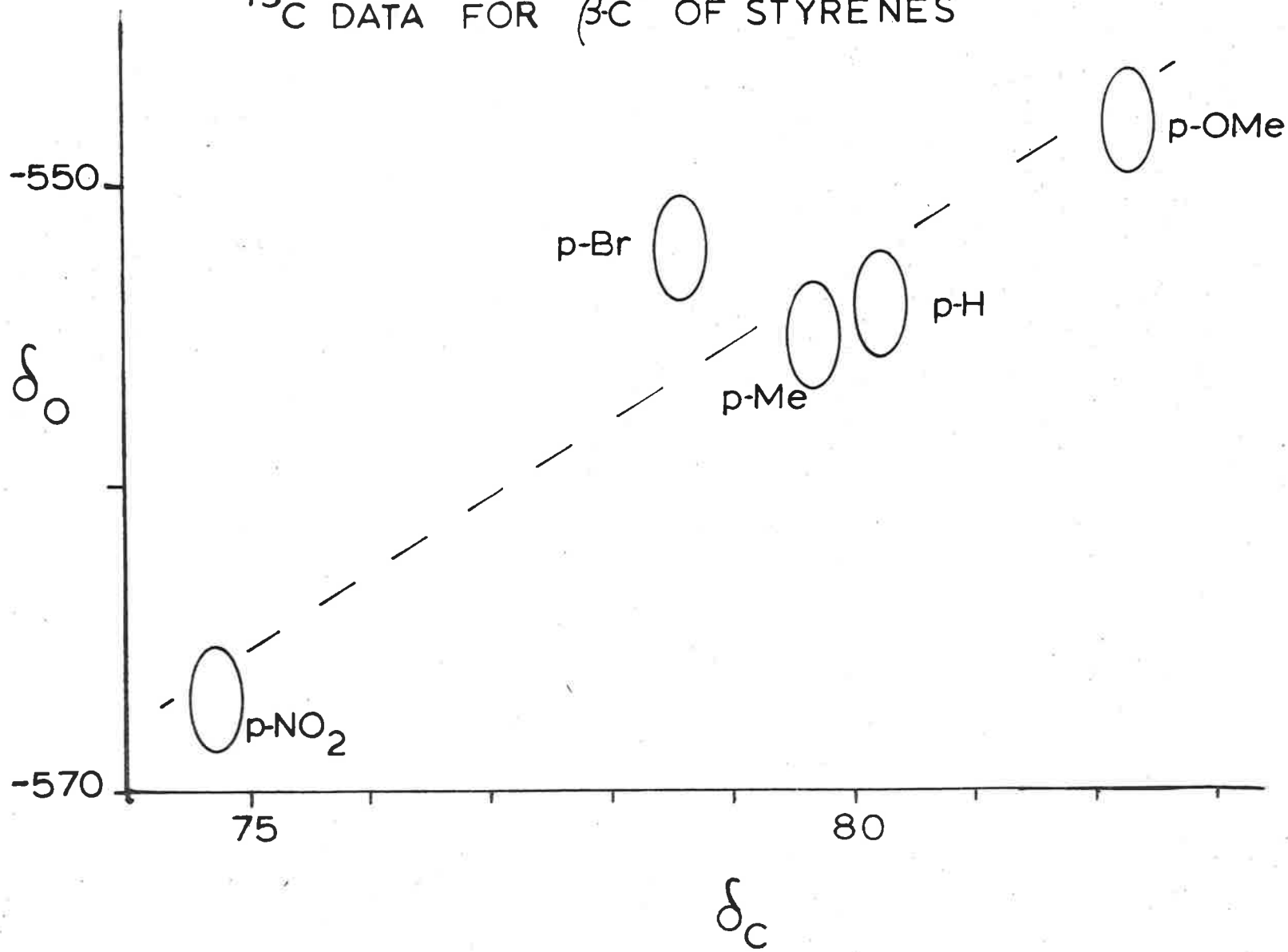
Jake

J. B. Stothers
Professor of Chemistry

JBS:caf

^{17}O SHIFTS OF SOME ACETOPHENONES vs.

^{13}C DATA FOR β-C OF STYRENES



TATA INSTITUTE OF FUNDAMENTAL RESEARCH

National Centre of the Government of India for Nuclear Science and Mathematics

Telegrams: ZETESIS HOMI BHABHA ROAD, BOMBAY 5 Telephone: 213141

Dr. B.L. Shapiro
Department of Chemistry
Illinois Institute of Technology
Chicago, Illinois 60616

March 11, 1968

Dear Dr. Shapiro:

Thank you for your reminder about the Newsletter.

Some of our recent work has been concerned with the Nuclear Magnetic Resonance of non-magnetic atoms in paramagnetic compounds.

We have studied the ^{19}F NMR in a number of transitions metal fluorides i.e. FeF_3, CuF_2, $KFeF_3$, $RbFeF_3$ and K_2NiF_4. The temperature and field dependence of these shifts are correlated with their magnetic susceptibility data and results discussed in terms of the hyperfine interactions. In FeF_3, the shift shows an anomalous temperature dependence (rather largely independent!) as has been observed from the magnetic susceptibility measurements. The results are discussed in the light of antisymmetric coupling of the form $d. [S_1 \times S_2]$ which cants the sublattice magnetization and gives rise to weak ferromagnetism. In CuF_2, the shift has a maximum value at a temperature which is 42° above the T_N deduced from the susceptibility measurement. In both $KFeF_3$ and $RbFeF_3$, the ^{19}F shift follows the magnetic susceptibility and the resonance completely disappears below their respective Neel temperatures.

K_2NiF_4 is very interesting from its magnetic properties point of view, being the only compound of the kind reported so far which exhibit

a true two-dimentional antiferromagnetic layer structure. The ^{19}F NMR shift shows only a small temperature dependence in the region 450°K down to its T_N. The signal disappears below 228°K(It is 48° above the previously reported value of T_N = 180°K for this compound). It is likely that there is a residual long range order in this compound till 228°K. The detailed work on this is in progress.

Whereas the ^{19}F NMR in $RbFeF_3$ is shifted towards the lower field giving a positive spin density at the fluorine site, the ^{85}Rb and ^{87}Rb NMR shows a small shift towards high field, resulting in a negative spin density at the Rb site. We have also seen the ^{87}Rb resonance in the ordered state of this compound. All the results will be published shortly.

Yours sincerely,

R. Vijayaraghavan

R. Vijayaraghavan

K.R.K. Easwaran

K.R.K. Easwaran

Title

Nuclear Magnetic Resonance in some transition metal fluorides.

RVcjt

115-28

GROUPEMENT AMPÈRE

ATOMES ET MOLÉCULES PAR ÉTUDES RADIO-ÉLECTRIQUES

Genève, le 22 mars 1968

SECRÉTARIAT
Institut de Physique de l'Université
de Genève
32, boulevard d'Yvoy
1211 Genève 4 / SUISSE
℗ 24 12 68
Chèques postaux 12-15847

GJB/dm

Professeur B.L. Shapiro
Illinois Institute of Technology
Technological Center

Chicago 60616

U. S. A.

Cher Professeur Shapiro,

Merci pour votre rappel de la date limite de notre contribution.

XVème Colloque A.M.P.E.R.E. - Grenoble / septembre 1968

Le XVème Colloque A.M.P.E.R.E. se tiendra du 16 au 21 septembre 1968 dans les locaux de l'Université de Grenoble, sur le Domaine Universitaire de Saint-Martin d'Hères.

Le programme se rapprochera au maximum de la définition du sigle "A.M.P.E.R.E. : Atomes et Molécules Par Etudes Radio-Electriques". Il se décomposera en cinq rubriques fondamentales :

A - Etudes radio-électriques d'états atomiques.
B - Propriétés et relaxation diélectriques (essentiellement solides).
C - Résonance paramagnétique électronique.
D - Résonance paramagnétique nucléaire.
E - Etudes radio-électriques de produits biologiques.

Les séances de travail auront lieu du lundi 16 au vendredi 20 septembre inclus, la réception des participants commençant le dimanche 15 septembre, et le samedi 21 étant réservé à des visites de laboratoires.

Les séances seront consacrées à :

1°) Des exposés invités.
2°) Des communications présentées en séances formelles.
3°) Des communications discutées en "tables rondes".

Les textes des communications doivent parvenir aux organisateurs avant le 15 juin 1968 pour être soumis au Comité de lecture. Ce Comité classera les textes retenus. Les textes complets des communications présentées en séances formelles, ainsi que les résumés des communications discutées en tables rondes seront publiés.

Les auteurs des communications soumises seront avertis dans la seconde quinzaine de juillet des décisions du Comité de lecture. Les épreuves des textes ou résumés retenus pourront être corrigées pendant le déroulement de la Conférence. La publication des comptes rendus est prévue pour la fin de l'année 1968.

Manuscrits

Les textes ne doivent pas dépasser deux mille mots (quatre pages imprimées); chaque photographie, schéma ou table doit être compté pour deux cent cinquante mots. Les résumés doivent être tapés dans le cadre d'une seule des feuilles spéciales jointes à cette circulaire. Ils ne doivent pas dépasser deux cent cinquante mots.

Les textes et résumés doivent être rédigés en français ou en anglais, langues officielles du XVème Colloque A.M.P.E.R.E.

Les auteurs doivent garantir que les textes proposés n'ont été, ni publiés, ni soumis pour publication à d'autres éditeurs. Dans le cas où un texte aura été retenu pour être présenté en séance, son matériau ne devra pas être soumis à une autre publication pendant l'année qui suivra le Colloque.

Les auteurs paieront à l'éditeur un droit de 50 florins par page imprimée, exception faite, toutefois, pour les deux premières pages de chaque article.

Inscriptions

Date limite : 15 juin 1968

Les droits d'inscription s'élèvent à 100 francs français par participant scientifique, et à 30 francs français pour toute personne accompagnante. Seuls les participants scientifiques recevront gratuitement le volume des comptes rendus. Les droits doivent parvenir aux organisateurs en même temps que l'inscription. Ils ne comprennent ni les frais d'hébergement, ni ceux de nourriture.

Hébergement

L'hébergement pourra avoir lieu soit en Maison d'étudiants, sur le Domaine Universitaire (chambres individuelles seulement), soit en hôtel à Grenoble (tarifs joints). Des services d'autobus assurent une liaison rapide entre le Domaine Universitaire et la ville. Les repas pourront être pris au Restaurant Universitaire, sur le Domaine.

Des circuits touristiques sont prévus pour les jours qui suivent la Conférence.

Je vous enverrai très prochainement une nouvelle lettre sur les travaux en cours de notre laboratoire.

Je vous prie de croire, cher Professeur, à l'assurance de mes meilleurs sentiments.

Prof. G.J. Béné

WAYNE STATE UNIVERSITY

COLLEGE OF LIBERAL ARTS DETROIT, MICHIGAN 48202

DEPARTMENT OF CHEMISTRY

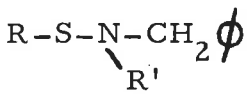
March 29, 1968

Professor Bernard L. Shapiro
Department of Chemistry
Stanford University
Stanford, California 94305

Chemical Shift Nonequivalence of Diastereotopic Protons in Sulfenamides

Dear Professor Shapiro,

We recently reported that N, N-dibenzyl trichloromethanesulfenamide(Ia) exhibits chemical shift nonequivalence of the diastereotopic benzyl methylene protons.[1] We have now completed a study of the variable n.m.r. spectra of I b-d and II a-b. As with Ia



I a $R = CCl_3$, $R' = CH_2\phi$
b $R = CCl_3$, $R' = CH_3$
c $R = CCl_3$, $R' = CH_2CH_3$
d $R = CCl_3$, $R' = CH(CH_3)_2$

the spectra (in $CHCl_3$) of I b-d all exhibit AB quartets for the benzyl methylene hydrogens at temperatures below about 10°C. Nonequivalent ethyl methylene protons are also observed in I c. Upon raising the temperature the quartets broaden and coalesce. The pertinent data include free energy of activation for the process leading to coalescence are recorded in Table I.

We have also looked at the effect of solvent (Table II). It may be noted that while a solvent effect on chemical shift differences is apparent the height of the barrier is insensitive to solvent changes (at least for those solvents observed). Apparently solvation changes in going from ground state to transition state are not very important.

1. M. Raban, Chem. Communication, 1017 (1967).

Although we can not be sure about the conformational changes which give rise to coalescence, two alternatives suggest themselves:

1. Slow inversion of the nitrogen pyramid, and
2. Slow rotation around the sulfur nitrogen bond of the kind long known to exist in disulfides. The ground state in this case would be a nonplanar structure in which the C-S bond would be staggered with respect to the C-N-C angle. The transition state would correspond to a structure in which the CCl_3 group is eclipsing (or nearly so) one of the alkyl groups on nitrogen.

Precident favors the first explanation. Numerous examples of slow nitrogen inversion due to a heteroatom directly bonded to nitrogen have been reported recently. However, one would expect steric accelleration on the basis of the slow inversion model. The results in Table I indicate steric decelleration which is more in accord with the slow rotation model.

We are continuing our investigation of conformational processes in sulfenamides and hope to obtain more definitive evidence concerning the process which corresponds to exchange of diastereotopic nuclei in these systems.

I hope this contribution will suffice as the fee for a new subscription to IITNMRN.

Sincerely yours,

Morton Raban

Morton Raban

George Kenney

George Kenney

TABLE I - Steric Effects

Compound	$\Delta\nu$ (in Hz)	J (in Hz)	Tc	$\Delta G^{\ddagger}$ (in kcal/mole)
Ib	8.9	14.7	15°C	14.4
Ic	7.6	15.0	23°C	14.9
Ia	8.0	14.8	28°C	15.3
Id	5.4	15.4	47°C	15.9

TABLE II

Solvent Effect on the N. M. R. Spectra of Id

Solvent	$\Delta\nu$ (in Hz)	J (in Hz)	Tc	$\Delta G^{\ddagger}$ (in kcal/mole)
$CDCl_3$	5.4	15.4	47°C	15.9
Benzene	15.5	15.0	43°C	15.8
Pyridine	8.5	15.4	51°C	16.1
Hexane	7.3	15.0	43°C	15.8

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36.30.02

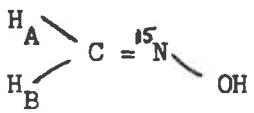
STRASBOURG, le 25 Mars 1968
1, rue Blaise PASCAL

Dr. B.L. SHAPIRO
Department of Chemistry
Illinois Institute of Technology
CHICAGO, 60616
Illinois
U.S.A.

Dear Barry,

Relative Signs of ^{15}N,H coupling constants in ^{15}N-formaldoxime.

In the course of our work on N,H coupling constants we prepared a sample of ^{15}N-formaldoxime (97% enriched) in water.



The p.m.r. spectrum of the CH_2 protons is shown in the figure. It is the AB part of an ABX spectrum. The A and B protons give respectively rise to the high field and low field quartets. The two AB sub-spectra may be identified from the relative intensities of the signals ; they respectively comprise lines 1-3-7-8 and 2-4-5-6. This assigument has been confirmed by double irradiation spin-tickling experiments. The chemical shift difference $\Delta\nu_{AB}$ may be extracted from the spectrum of the ^{14}N isotopomer. The analysis of this spectral pattern is straightforward. Agreement between the calculated and the experimental spectrum is only reached when opposite signs are assigned to the H_A-C=N and H_B-C=N couplings.

../..

../..

The spectral parameters are (water solution ; see Figure) :

$\delta(H_A) = 6.65$ ppm

$\delta(H_B) = 7.15$ ppm (from internal $(CH_3)_3Si(CH_2)_3SO_3N_a$)

$^2J_{H_AH_B} = 7.60$ H_z

$^2J_{NH_A} = \mp 13.88$

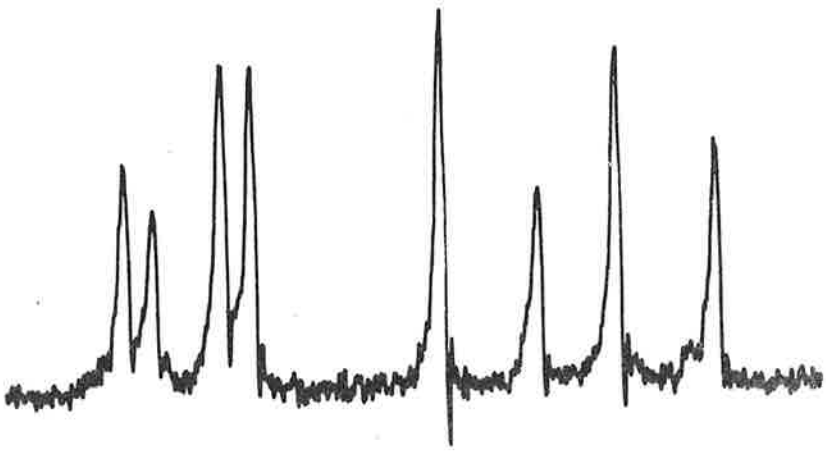
$^2J_{NH_B} = \pm 2.68$

Studies of other aldoximes in acid solution (CF_3COOD) have shown that in the corresponding protonated forms the N,H_A and N,H_B are also of opposite sign. On protonation $^2J_{NH_A}$ decreases markedly (= ca. 2-3 Hz) while $^2J_{NH_B}$ is only slightly affected.

With our best wishes,

Cordially,

D. Crépaux J.M. Lehn



60 MHz p.m.r. spectrum of the CH_2 protons of ^{15}N-formaldoxime (97% enriched) in water .

CARNEGIE-MELLON UNIVERSITY

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